Relationship Between Volatile Compounds and Sensory Attributes of Olive Oils by the Sensory Wheel

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ABSTRACT: Sixty-five volatile compounds and 103 sensory attributes were evaluated in 32 virgin olive oil samples from three different Mediterranean countries. Volatile compounds were analyzed with a dynamic headspace gas-chromatographic technique by using a thermal desorption cold-trap injector. The sensory analysis was conducted by six panels composed of assessors from the United Kingdom, Spain, the Netherlands, Greece and Italy. Principal-components analysis of sensory attributes was used to construct a statistical sensory wheel that represents the whole virgin olive oil flavor matrix. This wheel is composed of seven sectors that show the basic perceptions produced by the oil: green, bitter-pungent, undesirable, ripe olives, ripe fruit, fruity and sweet. The boundaries of each sector were calculated from the circular standard deviation of its sensory attributes. The relationship between sensory and instrumental analysis was determined by projecting volatiles onto the sensory wheel. Correlations of each volatile with the first two components of the principal-components analysis were taken as its coordinates (x,y) in the sensory wheel. Volatiles took up the most appropriate place within the sector that corresponded with their perception, and often close to the sensory attributes that explained their sensory properties. A gas-chromatographic/sniffing method was applied to virgin olive oil samples to assess the aroma notes that corresponded to olive oil volatile compounds and to verify the relationships found by the sensory wheel procedure. Most (89%) of the volatiles were well classified. Use of the statistical sensory wheel as an appropriate method to relate volatile and sensory data was clearly demonstrated.
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KEY WORDS: Sensory attributes, statistical sensory wheel, virgin olive oil, volatiles.

Flavor is one of the most important qualities of foodstuffs and plays a major role in consumer acceptance. Sensory evaluation is generally considered to be the ultimate method to measure flavor quality of foodstuffs, because chemical or instrumental procedures lack the acuity of the human senses and the ability to integrate perceptions. In recent years, however, many attempts have been made to obtain more objective results by using volatile compounds analysis and correlation between instrumental and sensory data (1). Pilgrim and Schutz (2), Noble *et al.* (3), and Kuentzel and Bahri (4) tried successively to relate sensory perceptions with chemical components. These attempts were, in fact, pictorial illustrations of some basic flavor relations, in which the overall flavor perception, defined by a wheel, was divided into sectors of basic/pure flavor notes, surrounded by or linked with the chemical components responsible for them. The sectors, defining "pure" flavor notes, and their place on the wheel were determined solely by subjective expert opinions, which did not take into account the possible synergy and antagonism between sectors defined by sensory perceptions and/or chemical components.

Most attempts to correlate flavor scores and volatile content of vegetable oils have focused on evaluating the loss of quality in the product. Morrison *et al.* (5) found a high correlation between flavor intensity scores and pentane content in stored sunflower oils. Warner *et al.* correlated the levels of volatile compounds with flavor scores to predict flavor stability (6) and found good correlations in soybean oil between total volatiles and hexanal with differences in flavor quality and stability (7).

Virgin olive oil requires a different approach because it is extracted from the fruit of the olive tree without any subsequent refining. It has a complex flavor, which is appreciated internationally by gourmets and cherished by native consumers. Olive oil sensory quality is so important that the relevant European Communities (EC) regulation includes sensory evaluation (8).

Because synergy and antagonism processes between volatile compounds contribute to the sensory evaluation of virgin olive oils, it is of great interest to ascertain the relationships between sensory attributes and the volatiles responsible for them. An appropriate method would have to gather much information from sensory and chemical quantitation, to reveal the basic flavor notes in a large set of sensory attributes and the volatile compounds responsible for them.

One possible approach is the use of the virgin olive oil sensory wheel (9,10), which was designed by following robust methodology rather than subjective opinions (3,4). This statistical sensory wheel (SSW) clustered many sensory attributes that qualify virgin olive oil flavor into seven basic perceptions, so that SSW could be seen as an automatic translator of the semantic meanings of the sensory attributes evaluated by habitual and potential users of this foodstuff (9) or as a method to interpret the attitudes of potential users (10).

Because sensory attributes that qualify virgin olive oil flavor have already been checked by different sensory profiles (11), the projection of volatile compounds onto SSW can give

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a plausible explanation of the relationship between sensory attributes and volatile compounds. Thus, the main objective of the present paper was to analyze the relationship between the sensory attributes produced by the whole virgin olive oil matrix and its volatile compounds by using SSW. The results were compared with those of a gas-chromatographic/sniffing method to determine the volatile compounds responsible for the sensory attributes most valuable for habitual and potential users of this vegetable oil.

MATERIALS AND METHODS

Materials. The data set was made up of 16 virgin olive (*Olea europea L.*) oil samples from fruit harvested in two different years (n = 32) and collected from Greece (Heraklion, Crete), Italy (Bitonto, Puglia) and Spain (Córdoba and Jaén, Andalusia). The varieties—Arbequina and Picual from Spain, Coratina and Cima di Bitonto from Italy, and Koroneiki and Tzunnati from Greece—were selected because they are widely used in the bottled olive oil trade (12,13). Fruits were picked, in perfect sanitary conditions, at three stages of ripeness: unripe, normal ripeness and overripe. Oils were obtained, under the best conditions, by using three extraction systems, centrifugation, percolation, and pressing, and subsequently freeze-stored until the moment of analysis. Table 1 summarizes the basic characteristics of each sample.

Sensory evaluations. Six panels, constituted by assessors of different nationalities, Spanish (A), Italian (B & D), Greek (C), British (E) and Dutch (F), carried out the quantitative descriptive analysis (14) of the thirty-two samples. Panels A, B and C strictly followed the EC regulation (8), and the score for each attribute was the result of the overall gustatory-olfactory-tactile perception. The assessors of panels A and B were fully trained with more than five years experience in evaluating all types of olive oil (virgin, current and lampante), and they worked at research centers. Participants of panel C were habitual consumers of this foodstuff, working at an olive oil factory. Panels D, E and F were not run by the EC regulation (8) but followed the International Standards Organization (ISO) document *General Guidance for Establishing a Sensory Profile* (15). Assessors of panel D were trained with mixtures of different types of olive oil and were students at an Italian University. British assessors (panel E) were trained with different oils (e.g., sunflower, nut, sesame, and olive), whereas the assessors of Dutch panel F were trained by evaluating different olive oil brands. Neither the British nor Dutch assessors had any previous experience in evaluating virgin olive oils. Table 2 summarizes the basic characteristics of the panels. Sample presentation was fully randomized, and all evaluations were done in triplicate. The assessors of each panel evaluated sensory attributes by the perceptions suggested by their panel leaders. The perceptions were flavor (a combination of olfactory-gustatory-tactile and kinaesthetic sensations), aroma (sensations perceived indirectly by the olfactory organ when tasting olive oil), odor (combination of sensations perceived directly through the nose), taste (sensations perceived when the gustatory papillae are stimulated), mouth feel (sensations perceived when a food comes into contact with all sensitive areas of the mouth) and after mouth feel/aftertaste (combination of sensations perceived after the stimulus has disappeared from the mouth).

Overall acceptability and sensory attributes associated with olive oil defects. Assessors of panels A, B, and C were also taught to rate overall acceptability (overall grading), associated with the complete profile sheet (8), on a 9-point scale. This evaluation was done after the assessors had completely evaluated the sensory attributes described on the profile sheet, by following easy rules built up through the experience of the trainers (16). The profile sheet of the EC regulation (8) is divided into two types of sensory attributes, "positive" and "negative." The latter clusters sensory attributes that indicate defectiveness and even unpleasantness: fusty, metallic, muddy sediment, mustiness, rancid, rough, sour, vinegary and winey. Virgin olive oil is required to have received a panel score ≥ 5.5 on the scale, with the implication that the higher the score, the better the quality (17). Assessors restrict the highest values of this panel score to those olive oils without sensory defects (8); thus the score of every "negative" attribute has to be low. The statistical analysis section describes how these "negative" attributes, evaluated by A,B,C, and F panels, were clustered into just one principal component for each panel, under the generic name of undesirable attribute because it gathers only "negative" attributes.

The sensory attributes are numbered in Table 3; these numbers (codes) will be used throughout this paper when referring to attributes.

Gas-chromatographic volatile analysis. Volatile compounds were analyzed with a dynamic headspace technique under determined optimized conditions as previously described (18). A 0.5-g olive oil sample was heated at 40°C and swept with N_2 (200 mL/min) for 15 min. Tenax TA (Chrompack, Middleburg, The Netherlands) was used as a trap. Volatiles were thermally desorbed at 220°C onto a fused-silica trap, cooled at −110°C for 5 min just before injection, accomplished by flash heating of the cold trap at 170°C for 5 min. The volatiles were transferred onto a fused-silica Supelcowax 10 capillary column (60 m, 0.32 mm i.d., 0.5 μm film thickness) (Supelco, Bellefonte, PA). The oven temperature

TABLE 1
Characteristics of Virgin Olive Oil Samples: Variety, Maturity and Extraction Systems

Variety	Maturity[a]	System[b]
Koroneiki	U,N,O	C
Koroneiki	N	P
Tzunnati	N	C
Coratina	U,N,O	C
Coratina	N	E
Cima di Bitonto	N	C
Picual	U,N,O	C
Arbequina	U,N,O	C

[a]U (unripe), N (normal ripeness), O (overripe).
[b]C (centrifugation system), P (percolation system), E (expression system).

TABLE 2
Basic Characteristics of the Panels That Carried Out the Sensory Evaluations

Group designator	A	B	C	D	E	F
Nationality	Spanish	Italian	Greek	Italian	British	Dutch
Number of assessors	10	10	14	11	9	8
Assessors' level[a]	F	T	T	F	T	T
Consumer[b]	H	H	H	H	P	P
Number of attributes	15	14	14	10	13	59
Scale[c]	S	S	S	S	U	U
Scores	1–5	1–5	1–5	1–9	100 mm	130 mm

[a]F (full), T (trained for this work).
[b]H (habitual), P (potential).
[c]S (structured), U (unstructured).

was held at 40°C for 4 min and programmed to rise at 4°C/min to a final temperature of 240°C where it was held for 10 min. A Hewlett-Packard 5890 series II gas chromatograph (Palo Alto, CA), fitted with an FID detector, was employed. For quantitative analysis, isobutyl acetate was used as internal standard. Volatiles were analyzed in duplicate.

Peaks were identified by mass spectral analyses with an MS 30/70 VG mass spectrometer (VG Analytical, Manchester, United Kingdom) and a VG 11/250 data system. Operating conditions were as previously described (19). Sample components were verified by comparison of mass spectral data with those of authentic reference compounds. When standards were not available, sample components were tentatively identified by mass spectrum matching with the National Bureau of Standards mass library collection. Table 4 shows all volatiles used in this study and their approximate mean concentration in samples.

Sensory properties of volatile compounds. To assess the aroma notes that correspond to olive oil volatile compounds, a high resolution gas chromatography (HRGC)/sniffing technique was applied to virgin olive oil samples of each variety (19). The effluent of the GC column was split 1 to 10 to the detector and the sniffing port, respectively. The odor-active regions of the eluate were evaluated, and their aroma notes were assigned by five assessors, two with more than ten years experience and three who were habitual consumers of virgin olive oil. The odor descriptions were noted on a form with a preprinted time scale, and assessors did not see the chromatogram. Assessors basically agreed on the odors of volatiles, although different semantic terms may have been used. A consensus-building discussion was held with assessors to decide the final sensory descriptors. The fourth column of Table 4 shows the characterization of the volatile compounds by sniffing.

The same assessors also carried out the smelling and tasting, in duplicate, at room temperature of four pure volatile compounds to assess their sensory properties. These compounds included ethyl acetate and hexan-1-ol (Merck, Darmstadt, Germany), (*E*)-3-hexen-1-ol (Aldrich, Milwaukee, WI) and 6-methyl-5-hepten-2-one (Sigma, St. Louis, MO). Volatile compounds were previously diluted in water or paraffin oil, depending on their solubility, to the same approximate concentration as found in virgin olive oil samples. The tasting of these volatiles allowed their characterization as rough and bitter (ethyl acetate), astringent and bitter [(*E*)-3-hexen-1-ol and 6-methyl-5-hepten-2-one] and rough (hexan-1-ol).

Data manipulation. Gas-chromatographic data were entered into a personal computer, and ASCII files were manipulated by a Fortran program to eliminate unwanted information from the chromatographic reports. The automated program performed the selection of peaks based on retention time ranges after visual recognition of a standard chromatogram. Retention time and areas of selected peaks, including the internal standard, were stored in a database (Ultrix/SQL, version 2.0)(20). Ratios of each of the selected peak areas to the area of the internal standard were used for statistical analysis.

The value of each sensory attribute was calculated first from the means of the triplicate evaluations of each attribute made by the assessors of each panel and then from the mean of assessors for each attribute. This process was carried out for each panel independently.

Statistical analysis. The statistical library used was Biomedical Computer Program (BMDP) (version 7.0)(21). The information for each sample, consisting of gas-chromatographic data and sensory attributes, was checked for skewness. The log transformation of standardized peak areas from a chromatogram or sensory attribute was considered a variable for a given virgin olive oil sample. All variables were standardized by Z-scores because there was a great difference between values of volatiles and sensory attributes.

Multivariate studies of different olive oils, evaluated by following the EC regulation (8), have demonstrated (9,16) that the "negative" attributes (fusty, metallic, mustiness, muddy sediment, rancid, rough, sour, vinegary, winey) were always plotted close to each other for virgin oils. Their closeness was due as much to their negative correlations with the other attributes ("positive" attributes) as to their low values. The histograms of these attributes showed that their values were low and sometimes almost zero. A simple analysis of their values confirmed this; for example, only two out of all "negative" attributes of panels A–C had a mean value greater than 0.5 on a 5-point scale.

For habitual users (panels A,B,C), all "negative" attributes (fusty, metallic, muddy sediment, musty, rancid, rough, winey) of each panel were reduced to just a principal component, under the generic name of undesirable attribute (numbers 8,17,25) by using principal-components analysis (PCA)

TABLE 3
Sensory Attributes Evaluated by the Panels with Different Methods of Perception

Panel[a]	Attribute[b]	Perception[c]	Code[d]	Panel	Attribute	Perception	Code
A	Olive fruity (green)*	Flavor	1	F	Twig	Odor	53
A	Apple*	Flavor	2	F	Pine/Harshy	Odor	54
A	Other ripe fruits*	Flavor	3	F	Lemon	Odor	55
A	Green*	Flavor	4	F	Orange	Odor	56
A	Bitter*	Flavor	5	F	Soft fruits	Odor	57
A	Pungent*	Flavor	6	F	Candies (fruit)	Odor	58
A	Sweet	Flavor	7	F	Wild flowers in springtime	Odor	59
A	Undesirable*	Flavor	8	F	Fermenting fruit	Odor	60
A	Olive fruity (ripe)*	Flavor	9	F	Farm	Odor	61
B	Olive fruity (ripe and green)*	Flavor	10	F	Oil for salads (soybean oil)	Odor	62
B	Other ripe fruits	Flavor	11	F	Tallow	Odor	63
B	Green*	Flavor	12	F	Cod liver oil	Odor	64
B	Bitter*	Flavor	13	F	Nuts	Odor	65
B	Pungent*	Flavor	14	F	Medicine	Odor	66
B	Sweet*	Flavor	15	F	Earthy	Odor	67
B	Allowable	Flavor	16	F	Taste intensity	Taste	68
B	Undesirable*	Flavor	17	F	Sweet	Taste	69
C	Olive fruity (ripe and green)*	Flavor	18	F	Salty	Taste	70
C	Apple*	Flavor	19	F	Olives	Taste	71
C	Other ripe fruits	Flavor	20	F	Green leaf	Taste	72
C	Green*	Flavor	21	F	Grass	Taste	73
C	Bitter*	Flavor	22	F	Green banana (not ripe)	Taste	74
C	Pungent*	Flavor	23	F	Dried green herbs	Taste	75
C	Sweet*	Flavor	24	F	Minced pepper	Taste	76
C	Undesirable*	Flavor	25	F	Red chili pepper	Taste	77
D	Tomato*	Aroma	26	F	Cream/butter	Taste	78
D	Ripe black olives*	Aroma	27	F	Coconut	Taste	79
D	Green olives*	Aroma	28	F	Caramel	Taste	80
D	Cut green grassy*	Aroma	29	F	Grotty	Taste	81
D	Artichoke*	Aroma	30	F	Velvet-like	Mouth feel	82
D	Apple*	Aroma	31	F	Sticky	Mouth feel	83
D	Yeast*	Aroma	32	F	Slightly burned/toasted	Taste	84
D	Bitter*	Taste	33	F	Ash tray	Taste	85
D	Pungent*	Mouth feel	34	F	Glue with ethyl acetate	Taste	86
D	Astringent*	Mouth feel	35	F	Refinery	Taste	87
E	Strength of olive	Odor	36	F	Bitter	Taste	88
E	Strength of olive	Flavor	37	F	Astringent	Mouth feel	89
E	Banana skins*	Flavor	38	F	Green	Aftertaste	90
E	Tomato*	Flavor	39	F	Fruity	Aftertaste	91
E	Sweet*	Odor	40	F	Cooling/evaporating	After mouth feel	92
E	Hay/composty*	Flavor	41	F	Glue with ethyl acetate	Aftertaste	93
E	Perfumey	Odor	42	F	Cocoa butter/white choc.	Aftertaste	94
E	Perfumey	Flavor	43	F	Putty/linseed oil	Aftertaste	95
E	Grassy*	Flavor	44	F	Used frying oil	Aftertaste	96
E	Almond*	Flavor	45	F	Trany	Aftertaste	97
E	Throatcatching*	Mouth feel	46	F	Dry wood	Aftertaste	98
E	Thickness	Mouth feel	47	F	Dusty	Aftertaste	99
E	Pungent	Flavor	48	F	Dry	After mouth feel	100
F	Odor intensity	Odor	49	F	Sharp/etching	After mouth feel	101
F	Sea breeze on the beach	Odor	50	F	Pungent/sharp throat	After mouth feel	102
F	Prickling	Odor	51	F	Undesirable	Flavor	103
F	Apple	Odor	52				

[a]Panel types are described in Table 2.
[b]Attributes evaluated by each panel. *Indicates terms selected for building the initial statistical sensory wheel.
[c]Kind of perception used to evaluate the attributes.
[d]The codes identify the attributes described in the paper.

(variance explained: 60.5% panel A, 64.5% panel B, and 52.8% panel C). The undesirable attribute numbered 103 was obtained by applying the same statistical procedure to "negative" attributes (rancid, rough, metallic, sour, vinegary) evaluated by potential users of panel F (variance explained: 54.4%). PCA allowed recognition of the contribution of each "negative" attribute to each one of the generically named undesirable attributes. Thus, rancid flavor contributed to unde-

TABLE 4
Volatile Compounds Identified and Quantified in the Olive Oil Samples. Sensory Characterization of Volatiles by HRGC Sniffing and the Statistical Sensory Wheel

Code[a]	Chemical compound	Concentration[b]	Sniffing[c]	Sensory wheel[d]
1	Methyl acetate	12.0		Green
2	Octene	11.0	Solvent-like	Green (50,91)
3	Ethyl acetate	91.2	Sweet, aromatic	Undesirable (25)
4	Butan-2-one	3.7	Fragrant, pleasant	Fruity
5	3-Methylbutanal	52.0	Sweet, fruity	Ripe fruit (3,7)
6	1,3-Hexadien-5-yne	16.7		Green (38)
7	An alcohol	48.3	Sweet, apple	Fruity (52)
8	Ethylfuran	56.3	Sweet	Miscellany 4 (sweet-green)
9	Ethyl propanoate	47.3	Sweet, strawberry, apple	Miscellany 4 (sweet-green)
10	An alcohol + hydrocarbon	222.1	Pungent acid	Miscellany 3 (ripe-undesirable)
11	3-Pentanone	0.3	Sweet	Green (91)
12	4-Methylpentan-2-one	0.3	Sweet	Green
13	Pent-1-en-3-one	204.5	Sweet, strawberry	Sweet (15)
14	2-Methylbut-2-enal	64.3	Solvent-like	Undesirable (54)
15	A hydrocarbon	228.8	Sweet, apple	Miscellany 4 (sweet-green)
16	Methylbenzene	0.1	Glue, solvent-like	Ripe fruit
17	2-Methylbut-3-enol	5.7		Undesirable
18	Butyl acetate	98.2	Green, pungent, sweet	Miscellany 4 (sweet-green)
19	Hexanal	178.2	Green, apple	Sweet
20	A hydrocarbon	139.9	Sweet, aromatic	Miscellany 4 (sweet-green)
21	2-Methylbutyl propanoate	6.3	Aromatic, ketone	Miscellany 2 (bitter) (85, 89)
22	2-Methyl-1-propanol	43.3	Ethyl acetate-like	Green (12)
23	(*E*)-2-Pentenal	16.0	Green, apple	Green
24	An alcohol	13.3	Greasy	Undesirable (8, 61)
25	(*Z*)-2-Pentenal	21.6	Green, pleasant	Miscellany 3 (ripe-undesirable)
26	Ethylbenzene	78.3	Strong	Bitter
27	(*E*)-3-Hexenal	58.3	Artichoke, green, flowers	Ripe fruit
28	(*Z*)-3-Hexenal	377.0	Green, green leaves, grassy	Green (79)
29	1-Penten-3-ol	599.4	Wet earth	Undesirable (61)
30	3-Methylbutyl acetate	55.3	Banana	Bitter (72,76)
31	Heptan-2-one	11.0	Fruity	Ripe fruit (16)
32	(*E*)-2-Hexenal	11479.2	Bitter, almonds, green-fruity	Bitter (72, 93, 45)
33	(*Z*)-2-Hexenal	38.3	Fruity, almonds	Green
34	2-Methylbutan-1-o1	23.0	Fish oil	Miscellany 3 (ripe-undesirable)
35	3-Methylbutanol	4.7		Undesirable
36	3-Methyl-2-butenyl acetate	25.6	Putty-like, unpleasant	Undesirable
37	Dodecene	99.9		Undesirable (25)
38	Pentan-1-ol	4.3	Pungent	Fruity (51)
39	Ethylbenzene	17.1		Fruity (2, 74)
40	Hexyl acetate	196.5	Sweet, fruity	Green (18)
41	A C_8 ketone	2.7	Fruity, mushroom-like	Green
42	Octan-2-one	8.3	Moldy	Ripe olives
43	3-(4-Methyl-3-pentenyl)furan	54.9	Paint-like strong	Ripe olives (41)
44	3-Hexenyl acetate	90.9	Green, green banana, green	Green (28, 29, 38)
45	(*Z*)-2-Penten-1-ol	518.5	Banana	Green
46	6-Methyl-5-hepten-2-one	27.6	Fruity	Bitter (88)
47	Nonan-2-one	24.6	Fruity	Miscellany 4 (sweet-green) (58)
48	Hexan-1-ol	722.9	Fruity, aromatic, soft	Undesirable (8, 61)
49	(*E*)-3-Hexen-1-ol	18.7		Miscellany 2 (bitter) (89)
50	Tridecene	166.5		Bitter
51	(*Z*)-3-Hexen-1-ol	604.4	Banana	Green
52	2,4-Hexadienal	1.3		Ripe fruit (7)
53	(*E*)-2-Hexen-1-ol	893.4	Green, grassy	Undesirable (32)
54	Acetic acid	29.0		Undesirable
55	Methyl decanoate	28.3	Fresh	Miscellany 1 (green-bitter) (62)
56	Hydrocarbon C_{11}	27.3	Varnish-like, pungent	Bitter
57	Hydrocarbon	579.8	Nail-varnish	Bitter-pungent (92)
58	2-Methyl-4-pentenal	22.0	Dried leaves	Bitter-pungent (36, 90)
59	1,2,4-Trimethylbenzene	20.7	Fish oil, unpleasant	Undesirable
60	4-Methyl-1-penten-3-ol	2.7	Sweety	Ripe fruit
61	Alcohol C_6 branched	4.3	Sweety	Ripe fruit
62	(*Z*)-2-hexen-1-ol	51.6	Green fruit	Green
63	2-Octenal	15.7	Fruity, soap	Green (1)
64	Propanoic acid	46.6	Aromatic, pungent	Miscellany 1 (green-bitter)
65	Hydrocarbon	205.1	Green	Green

[a]The codes identify the chemical compounds described in the paper.
[b]Mean concentration in µg/L.
[c]Sensory characterization of chemical compounds by HRGC/sniffing.
[d]The sector of the SSW where the chemical compounds was located. The numbers in parentheses are the codes of the attributes near the chemical compound.

sirable attribute numbered 8 (adjusted $R^2 = 0.94$), whereas rough after mouth feel contributed to undesirable attributes numbered 17 (adjusted $R^2 = 0.83$), 25 (adjusted $R^2 = 0.64$) and 103 (adjusted $R^2 = 0.71$).

Statistical sensory wheel (SSW). The SSW can be understood as a sensory representation of the flavor matrix of any foodstuff. This paper applies the procedure described by Aparicio and Morales (9). The applied methodology was as follows: (i) Calculation of the means of the triplicate evaluations of each attribute made by assessors and then the mean of assessors for each attribute. Thus, there was a value of each attribute for every sample. (ii) Selection of the sensory attributes, from the whole set of attributes (Table 3), based on their repeatability, to embody all of the terms expressed by the panels, with regard to sensory attributes described by the EC regulation (8). (iii) Detection of outliers with respect to the solution and among cases, applying Mahalanobis distance evaluated as χ^2/df. (iv) PCA of the selected attributes, without rotation. (v) Validation of the first two factors by applying cross-validation (22), repeated at least four times with different cancellation matrices. (vi) Plotting of selected attributes by the first two principal components. A circle of radius 1.0 is drawn at coordinates (0,0) onto this figure. (vii) Calculation of the limits of each sensory wheel sector. (a) Selection of the sensory attributes of each sector. The attributes should have similar semantic terms; for example, green perception clusters the attributes numbered 1 ,4, 10, 12, 18, 21, 28, 29, 38, 44. The selection can be made by cluster analysis (23) or taking into account the users' experience. (b) Radial projection of the coordinates of each attribute over the circle. Projection was made normalizing each coordinate by the Euclidian distance of the attribute from the origin. For example, given the coordinates of yeast $\alpha(0.40,-0.64)$, it is possible to calculate its Euclidean distance from the origin, $d = 0.76$, and then calculate its new coordinates (weighted coordinates) over the circle of radius 1, $W_\alpha = (0.53,-0.85)$. (c) Calculation of mean horizontal and vertical coordinates $W_{mean} = (W_{mean(\alpha x)}, W_{mean(\alpha y)})$ of each group is shown in Equations 1 and 2.

$$W_{mean(\alpha x)} = \frac{1}{n} \sum_{\alpha=1}^{n} W_{(\alpha x)} \quad [1]$$

$$W_{mean(\alpha y)} = \frac{1}{n} \sum_{\alpha=1}^{n} W_{(\alpha y)} \quad [2]$$

(d) Calculation of the length of the mean direction of each group. The length, R, was calculated according to the formula in Equation 3:

$$\bar{R} = \sqrt{\left(W_{mean(\alpha x)}\right)^2 + \left(W_{mean(\alpha y)}\right)^2} \quad [3]$$

(e) Calculation of the circular standard deviation index (S) by using Equation 4 for a two-dimensional space (9):

$$S = \sqrt{-2 \log_e \left(1 - \frac{1-\bar{R}}{0.293}\right)} \quad [4]$$

(f) Drawing a circle of radius S at the coordinates of W_{mean}. Arcs are constructed by drawing tangents to this circle from coordinates (0,0) and they define the sensory wheel sectors. (viii) Calculation of the correlations of each one of the rest of the sensory attributes with the first two factors of PCA. These values are taken as its coordinates (x,y) in this statistical sensory wheel. These attributes are drawn at the calculated coordinates.

The EC regulation (8) highlights the existence of clear groups of sensory perceptions that define each olive oil: green, pungent, bitter, fruitiness, ripe fruit, sweet and a miscellany of attributes for the assessment of presence of sensory defects.

Table 3 shows the sensory attributes selected by following the described methodology. No outliers with respect to the solution and among cases were detected. The largest squared multiple correlations (SMC) among the variables showed that "multicollinearity" and "singularity" were not a difficulty in these data sets. Cross-validation (22), repeated four times with different cancellation matrices, always indicated that the first two components were enough for this study.

Cluster analysis (Ward's method and city-block distance) was used to determine the basic sensory perceptions. PCA applied on the selected sensory attributes (Table 4) allowed definition of the basic perceptions and the attributes associated with them: undesirable (numbers 8, 17, 25, 27, 32), green (numbers 1, 4, 10, 12, 18, 21, 28, 29, 38, 44), bitter-pungent (numbers 5, 6, 13, 14, 22, 23, 33, 34, 35, 45, 46), ripe olives (numbers 9, 41), ripe fruit (numbers 3, 7, 11, 20), fruity (numbers 2, 19, 26, 31, 39) and sweet (number 15). Figure 1 shows the sensory wheel sectors obtained from the described methodology, once the other sensory attributes were projected.

Projecting volatiles onto the sensory wheel. Once the sensory wheel was built, the volatile compounds were projected onto it (Fig. 2). The correlations of each volatile compound with the first two components of PCA were taken as its coordinates (x,y) in this sensory wheel. Thus, all volatiles were standardized by the set of selected attributes, representing different kinds of consumers and taking into account the only existing regulation (8) on olive oil quality.

The position of volatiles and sensory attributes on the sensory wheel determines their information content. There is less information from data placed close to the center of the circle than from that placed close to the perimeter of the circle; hence, we can see the sensory wheel in terms of probability. Thus, the most noteworthy volatile compounds, in terms of basic contribution to virgin olive oil flavor matrix, are the following (Table 4): numbers 3, 5, 8, 9, 13, 15, 18, 19, 22, 24, 26, 27, 28, 29, 34, 37, 40, 44, 46, 48, 52, 53, 59, 60, 61, 63, 65.

On the other hand, the sensory wheel sectors do not cover the whole wheel—there are sectors that no perception qualifies clearly; these are the so-called miscellanies. They lie between sectors cleanly defined by one of the seven basic perceptions, and their existence is logical because a perception disappears gradually and not abruptly.

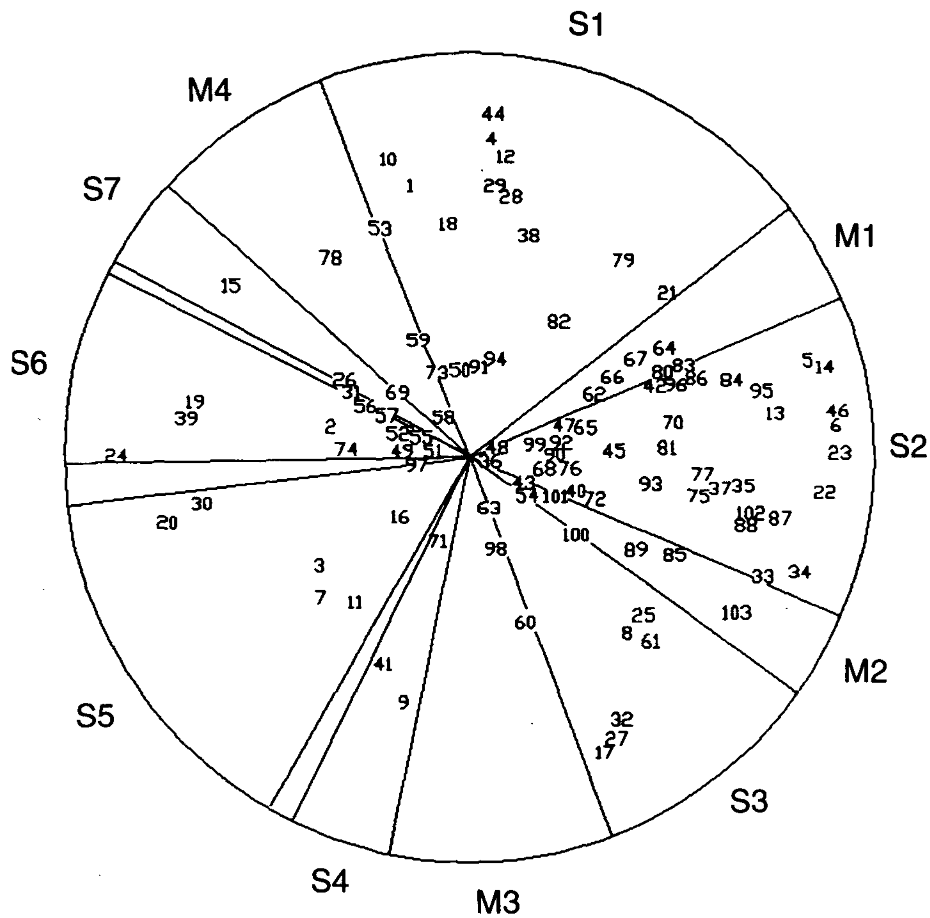

FIG. 1. Statistical sensory wheel of all sensory attributes evaluated by panels. Sensory wheel sectors were calculated by selected sensory attributes (Table 3) by following the described methodology. S1: green sector, S2: bitter-pungent sector, S3: undesirable sector, S4: ripe-olives sector, S5: ripe-fruit sector, S6: fruity sector, S7: sweet sector, M1: miscellany 1, M2: miscellany 2, M3: miscellany 3, M4: miscellany 4.

Statistical algorithms were run on a DEC-Station 5200, and figures were drawn by Autocad (release 12) (24).

RESULTS AND DISCUSSION

Because the statistical sensory wheel has been designated as the mathematical representation of the complex virgin olive oil flavor matrix, and volatile compounds have been described as responsible for virgin olive oil flavor, a procedure that gathered both datasets would show whether sensory wheel sectors are mathematical artifacts, or if they indicate that the volatiles are responsible for basic flavor perceptions of olive oil aroma.

The assessors, in qualifying a volatile by sniffing, search their cerebral "library" for the sensory attribute that best explains the odor. The mathematical process follows a similar path because the volatile "looks for" the set of attributes that best qualifies it in the whole set of attributes perceived in the oil. Figure 2 shows volatile compounds projected onto the sensory wheel, and Table 4 shows the results of projecting volatiles on the sensory wheel vs. the sensory attributes perceived by sniffing. Although mathematical equations cannot smell or taste chemical compounds, the results seem fairly accurate. There was disagreement between HRGC/sniffing and SSW on only six volatiles [octene (number 2), ethyl acetate (number 3), (*Z*)-2-pentenal (number 25), pentan-1-ol (number 38), hexan-1-ol (number 48) and (*E*)-2-hexen-1-ol (number 53)], 9.23% of sniffed volatiles. Another three volatiles were classified by their taste perception (bitter, pungent or astringent) rather than their odor [2-methylbutyl propanoate (number 21), 3-methylbutyl acetate (number 30), 6-methyl-5-hepten-2-one (number 46)].

The well-classified volatiles (89% of the total volatiles) were divided into three groups according to their place on the sensory wheel: (i) volatiles characterized by the sectors defining exactly the perception sniffed; (ii) volatiles classified by the attributes surrounding their place on the sensory wheel; (iii) volatiles classified by perception of their taste, mouth feel or after feel rather than odor.

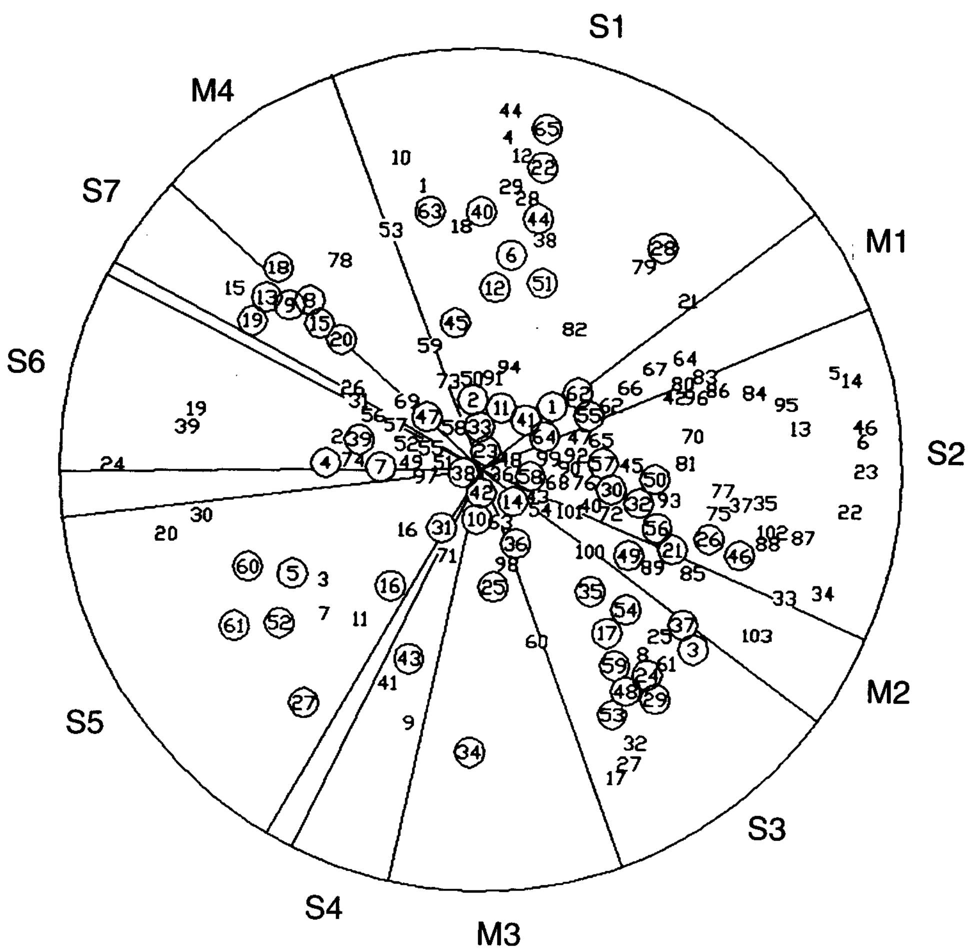

FIG. 2. Statistical sensory wheel with quantitative data of volatile compounds projected onto it. Volatile codes have been circled. Sectors and miscellanies are as in Figure 1.

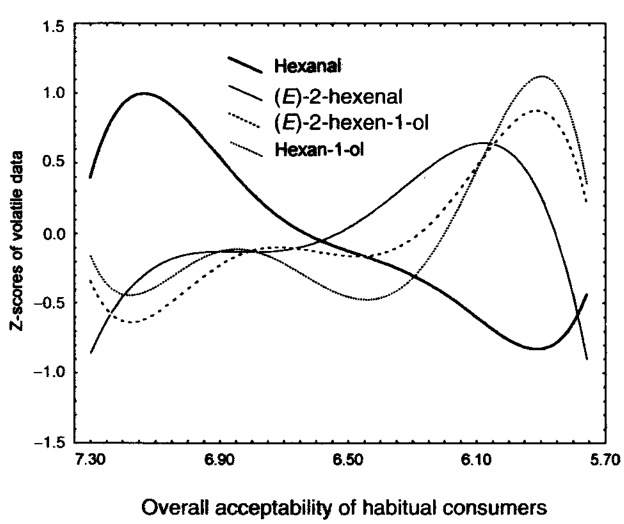

FIG. 3. Overall acceptability of habitual consumer assessors (on a 9-point scale) vs. Z-scores of quantitative data of volatile compounds, showing the different content of hexanal, (*E*)-2-hexenal, (*E*)-2-hexen-1-ol and hexan-1-ol in virgin olive oil samples.

Study of the different sensory wheel sectors. Sweet sector (S7). The EC regulation (8) defines sweet as "pleasant taste, not exactly sugary, but found in oil in which bitter, astringent and pungent do not predominate". Except for the sweet attribute evaluated by panel E (number 40), the other sweet attributes (numbers 7, 15 ,24, 69) agreed with the definition of the EC regulation, although the possible vagueness of this definition could explain why these attributes appear in different sectors of the SSW. The sweet sector, made up of attributes numbered 15 and 69, lies between the green and fruity sectors, whereas the sweet attributes (numbers 24, 7) evaluated by panel A,C appeared in the fruity and ripe-fruit sectors.

Two volatiles appeared in the sweet sector (Fig. 2) including pent-1-en-3-one (number 13), a ketone described as sweet, strawberry by sniffing, and hexanal (number 19) described as green, apple (Table 4). The latter compound, commonly associated with oxidation of vegetable oils, contributes considerably to the flavor of virgin olive oil (25). It appeared very near to the sensory attribute of "sweet" (number 15) (Fig. 2). The perception of some compounds varies with their concentration. Thus, high dilutions of hexanal are used to obtain fruity notes in perfumes (26). In virgin olive oils, the concentration of hexanal contributes to a particular sweet perception that is very pleasing to users. In fact, the samples of oil with a low assessment of acceptability contained lower amounts of hexanal than those with a higher assessment of acceptability (Fig. 3).

Miscellany 4 (M4). Close to those volatiles of the sweet sector and highly correlated with them ($R > 0.95$) was a group of compounds that are placed in miscellany 4 (which can be defined as sweet-green). Prominent among these are the esters, such as butyl acetate (number 18), and ethyl propanoate (number 9), furanic compounds, such as ethylfuran (number 8), and two hydrocarbons (number 15 and 20) (Fig. 2) tentatively identified as dimethyl nonadienes. Some nonadienes have been described sensorially as buttery (27), and these two compounds appear close to the attribute cream/butter (number 78). In this sector, with less information, nonan-2-one (number 47) also appears. All of these volatiles were described as sweet-fruity-green during sniffing (Table 4).

Fruity sector (S6). Next to the sweet sector is one we have called fruity (S6) (Fig. 1, 2), followed by a ripe fruit sector (S5). In fact, the two sectors can be considered a single, large-fruity sector ranging from green fruity to ripe fruity. The fruity sector includes the attributes sweet (number 24), tomato (number 39), and apple (number 19) (Fig. 1, Table 3), that signify a not very ripe fruit. Three volatiles with little information appear in this sector: a hydrocarbon (number 39), butan-2-one (number 4), and an alcohol (number 7) (Fig. 2). Volatiles number 7 and number 4 were described as sweet, apple and fragrant, pleasant (Table 4).

Ripe fruit sector (S5). In the ripe fruit sector appear (with greater information) the volatiles alcohol C_6 (number 61) (sweet), 4-methyl-1-penten-3-ol (number 60) (sweet), 2,4-hexadienal (number 52), (*E*)-3-hexenal (number 27) (artichoke), 3-methylbutanal (number 5) (sweet, fruity), and (with less information) methylbenzene (number 16) (glue, solvent) and heptan-2-one (number 31) (fruity) (Fig. 2, Table 4). The description from sniffing these compounds was, in general, fruity and sweet, corresponding to a ripe fruit. 2,4-Hexadienal was not detected by sniffing, although references describe it as fresh, floral (28) and oily and fatty (29), which could explain its being in this sector. The sector comprises the sensory attribute of other ripe fruits of the different panels (numbers 3, 11, 20), together with attributes such as sweet (number 7) and artichoke (number 30).

Ripe olive sector (S4). The next sector was also a ripe fruit sector but specific to the olive. It is defined by sensory attributes such as olive fruity (ripe) (number 9), compost (number 41), and olives (number 71) (Fig. 1, Table 3). 3-(4-Methyl-3-pentenyl)furan (number 43), and (with a lesser contribution) octan-2-one (number 42) were located in this sector. Both were described as moldy and paint-like strong by sniffing (Fig. 2, Table 4).

Miscellany 3 (M3). Next is the sector miscellany 3, with attributes of over-ripe, bordering on what could be considered undesirable in virgin olive oil. In this sector appear three volatiles (Fig. 2). The first is 2-methylbutan-1-ol (number 34), described as fish oil (Table 4). With less information appears (*Z*)-2-pentenal (number 25). Although those sniffing this compound described it as green, pleasant, it was placed in this sector because it could be classified by taste rather than smell; it appears close to the bitter-pungent group whose perceptions are of mouth feel and tasting. Third, there was a peak that was comprised of an alcohol plus a hydrocarbon (number 10), and described as pungent, acid.

Undesirable sector (S3). The next sector includes the undesirable attributes. Its presence in the evaluation is necessary because, in some cases, oils may present sensory defects. The samples analyzed in this study (high-quality virgin olive oil) did not show very high values for these attributes.

The undesirable sensory attributes (number 8, 17, 25), evaluated by standard panels (8), define this sector along with the attributes described as yeast, fermenting fruit, farm, tallow, dry wood and dry (numbers 32, 60, 61, 63, 98 and 100) (Fig. 1, Table 3). The Italian assessors (panel D) evaluated ripe olives (number 27) as undesirable.

Various volatiles are placed in this sector of the wheel. The most noteworthy (supplying the greatest information) are 1-penten-3-ol (number 29), (*E*)-2-hexen-1-ol (number 53), alcohol (number 24), hexan-1-ol (number 48), ethyl acetate (number 3), dodecene (number 37), 1,2,4-trimethylbenzene (number 59), 2-methylbut-3-enol (number 17), acetic acid (number 54), and 3-methyl butanol (number 35) (Fig. 2). As expected, most of these compounds are not present in high concentrations in the samples analyzed. Only hexan-1-ol, (*E*)-2-hexen-1-ol, and 1-penten-3-ol are major peaks in virgin olive oil headspace, and their concentration is lowest in the oils with the highest acceptability scores and highest in those with the lowest acceptability scores (Fig. 3). The sensory characterization of hexan-1-ol by sniffing, described as fruity, aromatic, soft (Table 4) disagrees with the sector "undesirable" in which it was placed. In tasting this volatile compound, however, assessors described a rough after mouth feel perception, which agrees with the main "negative" attributes that contribute to the undesirable sector, rancid (panel A) and rough (panels B,C). The sensory properties of (*E*)-2-hexen-1-ol, have been described as powerful, leafy, green and wine-like in the literature (28). These sensory properties agree with the results from sniffing (green, grassy) but disagree with the SSW sector (undesirable) where (*E*)-2-hexen-1-ol was located. Ethyl acetate was also described by assessors as having a rough and bitter aftertaste in the tasting, which completely agrees with its location in the undesirable sector.

These disagreements between HRGC/sniffing and SSW (volatiles coded 3, 48, 53) might be explained by the fact that sensory properties of volatile compounds can change with concentration and that new sensory properties can be achieved if other compounds are present, because of synergism, suppression and enhancement. Because SSW took into acount the sensory attributes evaluated in the whole virgin olive oil matrix, differences between sniffing and SSW results were expected.

Miscellany 2 (M2). Between the sectors undesirable and bitter-pungent lies miscellany 2, a diffuse zone that contains attributes close to both sectors, undesirable (number 103), ashtray (number 85), and astringent (number 89) (Fig. 1, Table 3). In this sector only two volatiles appear 2-methylbutyl propanoate (number 21) and (*E*)-3-hexen-1-ol (number 49) (Fig. 2, Table 4). The presence of the latter is noteworthy, because it is considered responsible for the green flavor of fruits. Nevertheless, it is placed in this sector with medium information because, although the odor of the compound is green, it contributes considerably to the taste of virgin olive oil. The taste was described as astringent and bitter, which is consistent with the description of its sensory characteristics in the literature as intensely green, bitter, fatty (28).

Bitter-pungent sector (S2). Virgin olive oil normally has a bitter flavor whose intensity varies depending on the olive variety. Most of the compounds responsible for bitterness in olives are phenolics. These behave as antioxidants, giving the oil stability to oxidation (30).

Bitter-pungent is the sector with the most attributes. Bitter (numbers 5, 13, 22, 33, 88), pungent (numbers 6, 14, 23, 34) and throatcatching (number 46) appear with greater information in the sector. The presence of some sensory attributes within the bitter-pungent sector, many of them evaluated by panel F, can be explained by their high correlation with different bitter-pungent attributes, such as refinery (number 87), glue with ethyl acetate (numbers 86, 93), green (number 90), nuts (number 65), slightly burned/toasted (number 84), and putty/linseed oil (number 95). Certain sensory notes are explained by their names, such as green leaf (number 72), red chili pepper (number 77), minced pepper (number 76), and intensity taste (number 68), but in some cases, such as frying oil (number 96) or sticky (number 83), we cannot offer either explanation for their presence or support for their exclusion. An interesting case is the attribute grotty—unpleasant or of poor quality—(number 81), which appears in this group, correlated with bitter (number 88) at $R = 0.80$, indicating that bitter-pungent-astringent perceptions are unpleasant for nonhabitual consumers, who are not accustomed to consuming virgin olive oils. For habitual consumers, these perceptions can also be quite unpleasant, depending on their intensity (31).

Few volatiles are found in this sector. This is logical because the sector is constituted by attributes of taste rather than smell (Table 4), and thus should include less volatile compounds. This sector is, therefore, the one of taste on the virgin olive oil sensory wheel. Thus, a hydrocarbon C_{11} (number 56) was characterized as pungent and ethylbenzene (number 26) as strong, whereas 6-methyl-5-hepten-2-one (number 46), whose sniffing suggested a fruity odor, was described in tasting as astringent-bitter; this is consistent with the sector in which it appears and with the attribute bitter (number 88), which is near by.

The other volatiles of this sector supply little information: 3-methylbutyl acetate (number 30), tridecene (number 50), a hydrocarbon (number 57), 2-methylpent-4-enal (number 58), and (*E*)-2-hexenal (number 32). The latter compound, also called "leaf aldehyde", is found in the highest concentration in the headspace of virgin olive oils. It was described by sniffing as green, bitter almonds, and appeared close to the attributes of almond (number 45), glue with ethyl acetate (number 93), sweet (number 40), and green leaf (number 72), which were found in this sector. This is in accord with the sharp, herbal-green, slight acrolein-like pungency notes used to describe *E*-2-hexenal by Bauer *et al.* 1990 (26). The concentration of this compound was highest in the oil samples with lowest acceptability (Fig. 3).

Miscellany 1 (M1). The next sector is miscellany 1, intermediate between the green and bitter-pungent sectors. It includes attributes such as sticky (number 83), earthy (number 67), and medicine (number 66). Only the volatiles methyl decanoate (number 55) and propanoic acid (number 64) appear with little information in this sector, close to the attribute salad oil (soybean oil) (number 62).

Green sector (S1). Last, is the green sector, which is one of the most important in the evaluation of virgin olive oils, both because it is a perception intimately linked to this foodstuff and because most of the compounds responsible for it are major volatiles in the virgin olive oil headspace. The green sector includes green and olive fruity perceptions (numbers 10, 21, 1, 18, 4, 12), plus cut green grassy (number 29), green olives (number 28), banana skins (number 38), grassy (number 44), grass (number 73), twig (number 53), seabreeze on the beach (number 50), with aftertastes of velvet-like (number 82), coconut (number 79), cocoa-butter (number 94), fruity (number 91), and wild flowers in springtime (number 59) appearing at the boundaries.

Most of the volatile compounds placed in this sector gave a sweet, green or fruit odor description when they were evaluated by sniffing. Green odor perception is the most remarkable, being produced mainly by C_6 aldehydes, alcohols and their corresponding esters, which are major components of the virgin olive oil headspace (32). Thus, 3-hexenyl acetate (number 44), (*Z*)-3-hexen-1-ol (number 51) and hexyl acetate (number 40) appear in the green sector close to sensory attributes such as banana skins (number 38), green olives (number 28), cut green grassy (number 29), and olive fruity (number 18), whereas (*Z*)-3-hexenal (number 28) is close to sensory attributes of green (number 21) and coconut (number 79). (*Z*)-2-hexen-1-ol (number 62) and (*Z*)-2-hexenal (number 33) appear with less information.

These characterizations agreed with the findings of other researchers. (*Z*)-3-Hexenal was described as green, leafy by Kuentzel and Bahri (4), and (*Z*)-3-hexen-1-ol as green leaves by Guth and Grosch (33) and leafy, green, grassy by Hatanaka (34). 3-Hexenyl acetate was associated with green odors, and hexyl acetate was described as fruity, pear-like by Bauer *et al.* (26). Other volatile compounds, contributing to the overall green perception with much information, included 2-octenal (number 63) (fruity, soap), 2-methyl-propan-1-ol (number 22) (ethylacetate-like) and a hydrocarbon (number 65)(green) and, with less information, 1,3-hexadien-5-yne (number 6), 4-methylpentan-2-one (number 12) (sweet), (*Z*)-2-penten-1-

TABLE 5
Regression Coefficients (*R*) of Applying Canonical Regression to Principal Components of the Sensory Attributes and Volatile Compounds of Each Sector. Total Variance Explained by Principal Components Selected by Cross-Validation

Sector	PC[a]	Sensory attributes Variance explained[b]	PC	Volatile compounds Variance explained	*R*
Green	5	82.9% (31.2, 27.5, 11.1, 6.8, 6.3)	5	82.8% (43.2, 13.4, 10.1, 9.2, 6.9)	0.84
Bitter	4	84.7% (41.7, 29.3, 9.6, 4.1)	2	69.0% (45.5, 17.5)	0.74
Undesirable	3	71.2% (35.3, 22.4, 13.5)	5	86.4% (46.8, 13.5, 12.3, 7.5, 6.3)	0.87
Ripe olives	2	70.6% (36.1, 34.5)	—[c]	100%	0.51
Ripe fruit	3	80.1% (35.6, 24.9, 20.5)	2	79.1% (54.2, 24.9)	0.73
Fruity	3	79.4% (45.2, 21.5, 12.7)	1	44.5%	0.62
Sweet	—[d]	100%	1	86.4%	0.71

[a]Number of principal components of each PCA after applying cross-validation.
[b]Total variance explained by principal components. Figures in parentheses are the variances explained by each principal component.
[c]There are no principal components because there was only one volatile compound.
[d]There are no principal components because PCA could not run with only two variables (numbers 15, 69).

ol (number 45) (banana), and (*Z*)-3-hexen-1-ol (number 51) (banana). From the location of the sensory attributes and volatiles in this sector, we may conclude that there are different kinds of green perceptions, ranging from "green, slightly bitter" to "green fruity" or, perhaps, to "green, sweet."

To check the mathematical goodness of fit of the statistical sensory wheel (Fig. 2), PCA was independently applied to all attributes and volatiles inside each sector. The factors of each PCA were selected by cross-validation. Table 5 shows the results of applying canonical regression (35) on the selected factors of each sector. The values of regression coefficients were higher than 0.70 for all sectors except for the ripe-olives sector ($R = 0.50$), which had only a volatile compound, and the fruity sector ($R = 0.62$) whose volatiles were so close to the center (0,0) that this sensory perception cannot be fairly explained by these volatiles.

In summary, the statistical sensory wheel can be seen as a sensory representation of virgin olive oil flavor because it absorbs the complex synergism/antagonism processes that are present in flavor perception. Projection of the volatile compounds onto this wheel allows us to understand the independent contribution of each compound to the total virgin olive oil flavor. The procedure permits us (i) to characterize the volatiles not only by sniffing but also by tasting; (ii) to point out some of the sensory attributes responsible for the basic perceptions but now take into account the possible synergy, suppression, or enhancement in the complex olive oil flavor matrix; and (iii) to explore a way to possibly substitute basic perceptions (sensory wheel sectors) for the group of volatile components classified within each sector.

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